

# (12) UK Patent Application (19) GB (11) 2 375 116 (13) A

(43) Date of A Publication 06.11.2002

(21) Application No 0206036.6

(22) Date of Filing 14.03.2002

(30) Priority Data

(31) 2001096699 (32) 29.03.2001 (33) JP

(71) Applicant(s)

Fujimi Incorporated  
(Incorporated in Japan)  
1-1 Chiryo, 2-Chome, Nishibiwajima-Cho,  
Nishikasugai-Gun, Aichi-Ken, Japan

(72) Inventor(s)

Noboru Yasufuku  
Toshiki Owaki  
Noritaka Yokomichi  
Junichi Hirano

(74) Agent and/or Address for Service

Mewburn Ellis  
York House, 23 Kingsway, LONDON, WC2B 6HP,  
United Kingdom

(51) INT CL<sup>7</sup>

C09G 1/02

(52) UK CL (Edition T )

C4V V131 V150 V190 V193 V2

(56) Documents Cited

GB 2354769 A

EP 1125999 A1

EP 0811665 A2

US 20010018270 A1

(58) Field of Search

UK CL (Edition T ) C4V

INT CL<sup>7</sup> C09G 1/02 , C09K 3/14

Online: PAJ, EPODOC, WPI

(54) Abstract Title

**A polishing composition comprising silicon dioxide, an oxidizing agent and an organic acid and its use in polishing a memory hard disk**

(57) A polishing composition for a memory hard disk, which comprises water, silicon dioxide, an oxidizing agent, and at least one organic acid selected from the group consisting of malic acid, maleic acid, lactic acid, acetic acid, citric acid, succinic acid, malonic acid, glycolic acid, adipic acid, ascorbic acid, itaconic acid, iminodiacetic acid, glyoxylic acid, formic acid, acrylic acid, crotonic acid, nicotinic acid, citraconic acid and tartaric acid, and which has a pH of at least 1 and less than 7 and contains substantially no metal ions. Preferably the silicon dioxide is in the form of colloidal silica and the oxidizing agent is hydrogen peroxide. A method of producing a memory hard disk comprises polishing a Ni-P disk with the polishing composition.

GB 2 375 116 A

2375116

- 1 -

POLISHING COMPOSITION AND METHOD FOR PRODUCING MEMORY  
HARD DISK BY MEANS THEREOF

The present invention relates to a polishing composition suitable for polishing the surface of a substrate in the production of the substrate for a memory hard disk i.e. a magnetic disk to be used for a memory device for computers, etc. More particularly, the present invention relates to a polishing composition to be used for the production of a memory hard disk represented by e.g. a Ni-P disk, a Ni-Fe disk, a boron carbide disk and a carbon disk, especially a polishing composition applicable to the production technique to obtain an excellent finished surface. Further, the present invention relates also to a method for producing a memory hard disk useful for a high capacity magnetic disk device having a high recording density, by means of the above polishing composition.

In recent years, along with the progress in downsizing and higher performance of computers, downsizing and larger capacities have been required for

magnetic disk devices as memory devices for such computers and for memory hard disks employed for such devices. The surface recording density on memory hard disks has been improved at a rate of a few tens% per  
5 year.

Accordingly, the space on a memory hard disk occupied by a predetermined amount of recorded information, is narrower than ever, and the magnetic force required for recording tends to be weak.  
10 Accordingly, in a recent magnetic disk device, it is required to minimize the flying height of the head, which is a space between the magnetic head for writing and reading information and the memory hard disk, and at present, the flying height of the head is reduced to a  
15 level of not higher than 1.0 micro inch (about 0.025  $\mu\text{m}$ ).

If there is a protrusion or waviness exceeding a certain height on a memory hard disk surface which is rotated at a very high speed, so-called "head crush" may  
20 occur, whereby the magnetic medium on the memory hard disk surface or the magnetic head may be damaged. Accordingly, it is necessary not to let a protrusion or waviness exceeding a certain height form on the memory hard disk surface.

25 On the other hand, a substrate for a memory hard disk (hereinafter referred to simply as a "substrate") which is most widely used at present, is one having an

electroless Ni-P plating film formed on a blank material. Here, the blank material is one obtained by fairing a substrate made of aluminum or other material by lathe processing by diamond turning, lapping by means of a PVA grinding stone prepared by fixing SiC grinding material, 5 or other methods, for the purpose of planarization.

So-called texturing may sometimes be carried out to impart concentric circular scorelines to the substrate after polishing, for the purpose of preventing sticking 10 of the magnetic head to the memory hard disk and preventing non-uniformity of the magnetic field on the memory hard disk due to scorelines in a certain direction different from the rotational direction of the memory hard disk, formed on the substrate surface by polishing. 15 Recently, for the purpose of further reducing the flying height of the head, light texturing is carried out wherein the scorelines formed on the substrate are further reduced, or a non-texture surface free from scorelines, is employed which is not subjected to 20 texturing.

If there is a scar (scratch) or a dent so-called a pit, which exceeds a certain depth or length, is present on the surface of a memory hard disk, it is likely that information can not completely be written in at such a 25 portion, thus leading to a defect of information so-called a "bit defect" or failure in reading the information, which causes an error of the magnetic disk

device. Here, with respect to the degree of the surface defect such as a scratch or a pit, even a shallow one will be problematic in a case where a memory hard disk is produced by using the above-mentioned non-texture

5 substrate. Even in a case where a substrate subjected to texturing or light texturing, is used, if there is one not removed by such texturing, the above-mentioned problem may result.

Accordingly, it is very important that in a  
10 polishing step i.e. in a preliminary step before forming a magnetic medium, roughness of the substrate surface is minimized to accomplish high planarization and smoothness and to suppress formation of scratches, pits, micro protrusions or other surface defects.

15 Heretofore, in polishing a substrate, it has been common to carry out finishing by one polishing step by means of a polishing composition (hereinafter sometimes referred to as a "slurry" from its nature) comprising aluminum oxide or other various abrasives, water and  
20 various polishing accelerators. However, by a single polishing step, it has been difficult to satisfy all of the requirements for removing waviness and relatively large surface defects such as protrusions or pits on the substrate surface and for minimizing the surface  
25 roughness within a certain period of time. Accordingly, a polishing process comprising two or more steps, has been studied.

In a case where the polishing process comprising two steps, the main purpose of the polishing in the first step will be to remove relatively large surface defects such as protrusions or pits and waviness on the substrate surface i.e. to improve planarization. Accordingly, a polishing composition is required which has a high ability of correcting the surface defects and waviness without forming deep scratches which can not be removed by polishing in the second step, rather than minimizing the surface roughness.

The purpose of polishing in the second step i.e. finishing or final polishing, is to minimize the surface roughness of the substrate. Accordingly, it is important that the polishing composition is capable of minimizing the surface roughness and improving the smoothness, rather than it has a high ability for correcting large surface defects or waviness, as required for polishing in the first step. Further, from the viewpoint of the productivity, it is also important that the stock removal rate is high. The degree of the surface roughness is determined depending upon the process for producing the substrate, the final recording capacity as a memory hard disk and other conditions. Accordingly, depending upon the degree of the desired surface roughness, a polishing process comprising more than two steps, may be employed.

Further, in recent years, various improvements have been made in processing a blank material by means of a

PVA grinder in order to reduce the processing cost. By such improvements, it is intended to reduce the surface roughness of a blank material, so that the quality such as the surface roughness and waviness of a plating substrate before polishing, is brought to a level after the conventional first polishing step. If such a processing is carried out, the conventional first polishing step will be unnecessary, and only so-called finish polishing will be required.

10 For the above purpose, particularly in finish polishing, polishing of a substrate has been carried out by means of a polishing composition prepared by thoroughly pulverizing aluminum oxide or other abrasive material to a proper uniform particle size, adding water  
15 thereto and then adding aluminum nitrate or various organic acids and other polishing accelerators, or a polishing composition containing colloidal silica and water.

However, when the former polishing composition is  
20 used, there has been a room for improvements from the viewpoint of formation of micro-protrusions or micro-pits. Whereas, when the latter polishing composition is used, there has been a room for improvements from the viewpoint of the stock removal rate, the roll off or dub  
25 off as an index of sagging of an end face of the substrate, or washing after the polishing.

In order to solve the above problems, JP-A-1-246068

discloses a specular finish method of an aluminum alloy substrate, wherein one side or both sides of an aluminum alloy substrate are polished by using, as a polishing agent, a colloidal silica having the pH adjusted to a level of at most 8 by an organic acid, and JP-A-2-185365 discloses a polishing method wherein an aluminum alloy disk comprising Mg and Mn, and the rest being Al and unavoidable impurities, is polished by using a colloidal silica solution having the pH adjusted to at most 8 by an organic acid.

Further, JP-A-10-204416 discloses a polishing composition for a memory hard disk comprising an abrasive and water, which further contains an iron compound dissolved in this composition. By using this polishing composition, a substrate having a small surface roughness can be obtained at a high stock removal rate.

Further, JP-A-11-167711 discloses a method for producing a magnetic disk substrate wherein polishing is carried out by means of a colloidal polishing agent containing at least 0.01 mol/l of trivalent iron ions in the form of a salt and containing silica particles having a particle size of at most 0.5  $\mu\text{m}$ .

Further, JP-A-2000-42904 discloses a slurry for surface polishing which comprises a liquid layer, an abrasive and species containing a ligand of an element contained in its surface, wherein the ligand is bonded to ions or atoms of various elements, and such bond is



sufficiently strong to remove the adsorbed ions or atoms of various elements from the surface.

However, as a result of the study by the present inventors, it has been difficult to satisfy all of  
5 requirements for a sufficient stock removal rate, suppression of scratches, prevention of corrosion and damage of the polishing machine and sufficient quality of the polished surface.

It is an object of the present invention to solve  
10 the above-mentioned various problems and to provide a polishing composition which has a high stock removal rate and which is capable of suppressing formation of scratches and capable of minimizing the surface roughness.

15 The present invention provides a polishing composition for a memory hard disk to solve the above problems, which comprises water, silicon dioxide, an oxidizing agent, and at least one organic acid selected from the group consisting of malic acid, maleic acid,  
20 lactic acid, acetic acid, citric acid, succinic acid, malonic acid, glycolic acid, adipic acid, ascorbic acid, itaconic acid, iminodiacetic acid, glyoxylic acid, formic acid, acrylic acid, crotonic acid, nicotinic acid, citraconic acid and tartaric acid, and which has a pH of  
25 at least 1 and less than 7 and contains substantially no metal ions.

Further, the present invention provides a method for

producing a memory hard disk, which comprises polishing a Ni-P disk having a surface roughness Ra of at most 30Å, by means of the polishing composition.

Still further, the present invention provides a  
5 method for producing a memory hard disk, which comprises preliminarily carrying out preliminary polishing at least once to obtain a substrate for a memory hard disk having a surface roughness Ra of at most 15Å and polishing the substrate for final polishing by means of the polishing  
10 composition.

The polishing composition for a memory hard disk of the present invention has a high stock removal rate and is capable of suppressing formation of scratches and capable of minimizing the surface roughness.

15 Further, by the method for producing a memory hard disk of the present invention, a memory hard disk having a small surface roughness can be obtained at a high stock removal rate while suppressing formation of scratches.

Now, the present invention will be described in  
20 detail with reference to the preferred embodiments.

#### Water

For the polishing composition of the present invention, any water selected from industrial water, city water, deionized water and distilled water, may be used  
25 so long as it contains substantially no metal ions. However, it is preferred to use one obtained by subjecting deionized water containing substantially no

metal ions to filtration to remove any foreign matters. For the polishing composition of the present invention, it is possible to use water containing metal ions, but, in such a case, it is necessary to remove metal ions from  
5 the final polishing composition by a suitable means such as ion exchange.

In the present invention, "metal" means elements of Group IA, Group IIA, Groups IB to VIIB and Group VIII.

#### Abrasive

10 The polishing composition of the present invention is characterized in that it contains silicon dioxide as an abrasive. Silicon dioxide includes various types differing in the nature and in the process for their preparation, such as colloidal silica, fumed silica and  
15 others. Among them, colloidal silica is preferred.

As a method for producing colloidal silica, it is common to employ e.g. a method wherein sodium silicate or potassium silicate is ion-exchanged to obtain ultra fine colloidal silica which is then subjected to grain growth,  
20 a method wherein an alkoxy silane is hydrolyzed with an acid or alkali, or a method wherein an organic silicon compound is heated and decomposed in a wet system.

Further, commercially available colloidal silica may have its pH adjusted by ion exchange or by addition of an  
25 acid and/or alkali, so that the colloidal state can be maintained even if the colloidal silica is in high concentration. Further, commercially available colloidal

silica includes one in a monodisperse state, one having several particles bonded to have an association ratio and one having a high purity having the content of metal impurities, etc. reduced. For the polishing composition  
5 of the present invention, any one of, such colloidal silica may be employed.

The particle size of the silicon dioxide is influential over the stock removal rate or the quality of the polished surface. With a view to maintaining a  
10 sufficient stock removal rate, sufficiently reducing the surface roughness of the polished surface and minimizing formation of scratches, the particle size of the silicon dioxide is usually from 0.005 to 0.2  $\mu\text{m}$ , preferably from 0.01 to 0.1  $\mu\text{m}$ , as the average particle size obtained  
15 from the specific surface area measured by a BET method.

The content of the silicon dioxide varies depending upon the type of the silicon dioxide to be used. However, with a view to maintaining a sufficient stock removal rate and maintaining uniform dispersibility and  
20 proper viscosity of the composition, the content of the silicon dioxide is usually from 1 to 40%, preferably from 1 to 20%, based on the total weight of the polishing composition.

#### Polishing accelerator

25 Further, the polishing composition of the present invention is characterized in that it contains an oxidizing agent. This oxidizing agent serves as a

polishing accelerator when used in combination with the after-described organic acid. In the present invention, the oxidizing agent is preferably one containing no metal ions. Such an oxidizing agent is preferably at least one  
5 member selected from the group consisting of chloric acid, perchloric acid, sulfuric acid, persulfuric acid, nitric acid, pernitric acid, hydrogen peroxide, iodic acid and periodic acid, particularly preferably hydrogen peroxide. The oxidizing agent may contain metal ions,  
10 but in such a case, it is necessary to remove metal ions from the final polishing composition by a suitable means such as ion exchange.

The content of the oxidizing agent in the polishing composition of the present invention varies depending  
15 upon the type of the oxidizing agent. However, with a view to obtaining sufficient chemical effects of the oxidizing agent thereby to maintain the stock removal rate and to prevent formation of scratches, the content of the oxidizing agent is usually at least 0.1%, based on  
20 the total weight of the polishing composition. On the other hand, from an economical viewpoint such that if the content exceeds a certain level, no further improvement of the stock removal rate can be obtained by a further increase of the oxidizing agent and with a view to  
25 preventing deformation or bursting of a storage container caused by oxygen generated by the decomposition of an excessive oxidizing agent, the content of the oxidizing

agent is usually at most 5%, preferably at most 3%, based on the total weight of the polishing composition.

Further, the polishing composition of the present invention is characterized in that it contains at least  
5 one organic acid selected from the group consisting of malic acid, maleic acid, lactic acid, acetic acid, citric acid, succinic acid, malonic acid, glycolic acid, adipic acid, ascorbic acid, itaconic acid, iminodiacetic acid, glyoxylic acid, formic acid, acrylic acid, crotonic acid,  
10 nicotinic acid, citraconic acid and tartaric acid. Such an organic acid serves as a polishing accelerator when used in combination with the above-described oxidizing agent, and it is particularly preferably at least one member selected from the group consisting of malic acid,  
15 maleic acid, lactic acid, itaconic acid and acetic acid. Further, a salt of an organic acid containing metal ions may serve to agglomerate silicon dioxide as abrasive, and the agglomerated particles may cause formation of scratches.

20 Further, the content of the organic acid in the polishing composition of the present invention varies depending upon the type of the organic acid. However, with a view to obtaining sufficient chemical effects of the organic acid to maintain a sufficient stock removal  
25 rate and suppressing formation of scratches, and from an economical viewpoint, the content of the organic acid is usually from 0.01 to 10%, preferably from 0.1 to 5%,

based on the total weight of the polishing composition.

Further, at the time of preparing the above polishing composition, various known additives may further be added for the purpose of quality maintenance or stabilization of the product, or depending upon the type of an object to be polished, processing conditions and other requirements for polishing.

Polishing composition

The polishing composition of the present invention may be prepared usually by mixing and dispersing the silicon dioxide, the oxidizing agent and the organic acid in water, and, if necessary, further dissolving other additives. The method for dispersing or dissolving such components in water, is optional. For example, they may be stirred by a vane type stirrer or dispersed by ultrasonic dispersion. Further, the order of addition of these components is also optional, and any component may be dispersed or dissolved first, or such components may be dispersed or dissolved simultaneously.

The polishing composition of the present invention may have the pH varied by addition of various additives. However, in order to obtain the effects of the present invention, the pH is required to be at least 1 and less than 7. Further, it is preferred that the pH is at least 2 and less than 4. By adjusting the pH of the composition within this range, a sufficient stock removal rate and the effect for preventing corrosion of the

polishing machine, etc., can be obtained. Accordingly, if the pH of the polishing composition is lower than 1 or 7 or higher, it is necessary to adjust the pH by adding an alkali or an acid. Even in such a case, it is

5 preferred to use an alkali or an acid containing no metal ions, so that the final polishing composition contains substantially no metal ions. If the raw material contains metal ions, it is necessary to remove the metal ions from the polishing composition by a suitable means.

10 The polishing composition of the present invention contains substantially no metal ions. The term "contains substantially no metal ions" means that the presence of metal ions in an amount not to impair the effects of the present invention, is allowable. Specifically, the  
15 content of metal ions in the polishing composition of the present invention is usually at most 500 ppm, preferably at most 50 ppm.

Further, for the storage of the polishing  
composition of the present invention, the polishing  
20 composition may be stored in a state where it is divided into two or more compositions, in order to prevent decomposition of the oxidizing agent. As an example, a method is conceivable wherein a slurry comprising the abrasive, the organic acid and water, is prepared and  
25 stored as a highly concentrated stock solution, and immediately before polishing or at the time of diluting the stock solution, the oxidizing agent is dissolved to



obtain a polishing composition having the predetermined composition. By employing such a method, it is possible to store the composition in a relatively high concentration for a long period of time.

5       The polishing composition of the present invention can be prepared, stored or transported in the form of a stock solution having a relatively high concentration, so that it is diluted for use at the time of actual polishing. The above-mentioned preferred concentration  
10 ranges of the respective components are those disclosed as the concentrations at the time of actual polishing. Thus, needless to say, if such a method of use is adopted, the composition would be a solution having a higher concentration during the storage or  
15 transportation. Further, from the viewpoint of handling efficiency, it is preferred that the polishing composition is prepared in such a concentrated form.

      The detailed mechanism is not clearly understood with respect to the reason why the polishing composition  
20 of the present invention is capable of providing a small surface roughness and a high stock removal rate and at the same time suppressing formation of scratches, in polishing a substrate. However, it may be explained as follows taking a Ni-P plated substrate as an example.

25       Firstly, with respect to the reason for the small surface roughness and high stock removal rate, it is considered that the Ni-P plated surface is etched by the

chemical action of the organic acid, and when the surface is oxidized and brittled by the chemical action of the oxidizing agent, a mechanical action by fine silicon dioxide particles is applied, whereby the Ni-P surface is  
5 readily removed in small units.

Further, with respect to the reason for suppression of formation of scratches, it is considered that the polishing composition of the present invention contains no metal ions, whereby agglomeration of silicon dioxide  
10 as abrasive will not be promoted.

#### Preparation of a memory hard disk

The method for preparing a memory hard disk according to the present invention, comprises polishing a memory hard disk by means of the above-mentioned  
15 polishing composition.

The substrate of the memory hard disk to be polished may, for example, be a Ni-P disk, a Ni-Fe disk, a boron carbide disk or a carbon disk. Among them, a Ni-P disk is particularly suitable.

20 The method for preparing a memory hard disk of the present invention may be used in combination with any conventional polishing method or polishing conditions for memory hard disks, so long as the above-described polishing composition is used.

25 For example, as the polishing machine, a single side polishing machine, a double side polishing machine or other machines may be employed. Further, the polishing

pad may be of a suede type, a non-woven type, a flocked type, a raising type or the like.

Further, the polishing composition of the present invention provides a small surface roughness, a high stock removal rate and is capable of suppressing formation of scratches. Accordingly, polishing can be carried out in one step on a substrate prepared to have a surface roughness  $R_a$  of at most  $30\text{\AA}$ , but may be carried out in two or more steps under different polishing conditions. In a case where the polishing process is carried out in two or more steps, it is preferred to make the polishing step employing the above-described polishing composition the final polishing step, i.e. a preliminarily polished substrate is polished by means of the polishing composition of the present invention. Further, in order to carry out the polishing by the polishing composition of the present invention more efficiently, it is preferred to have the surface roughness of the preliminarily polished substrate adjusted to  $R_a$  at most  $15\text{\AA}$  in the measuring method by means of an optical surface roughness meter.

Now, the polishing composition and the method for producing a memory hard disk, according to the present invention, will be described in further detail with reference to Examples. However, it should be understood that the present invention is by no means restricted by the constructions of the following specific Examples.

Preparation of polishing compositions

Firstly, colloidal silica (primary particle size: 0.035  $\mu\text{m}$ ), an oxidizing agent and an organic acid were added and mixed to deionized water, as disclosed in Table 1, to prepare test samples of Examples 1 to 32 and Comparative Examples 1 to 13. As hydrogen peroxide, an aqueous hydrogen peroxide solution having a concentration of 31% was used, but the content disclosed in Table 1 is the content as hydrogen peroxide. Further, the pH of each test sample was as shown in Table 1.

Preparation of substrates for polishing tests

Substrates were prepared to carry out evaluation of the above test samples by two step polishing. The polishing conditions were as follows.

Polishing conditions (first step)

Polishing machine: Double side polishing machine

Object to be polished: 3.5" electroless Ni-P substrate

Number of polished sheets: (two sheets/carrier)  $\times$  5 carriers  $\times$  2 batches = 20 sheets

Polishing composition: DISKLITE-A3510  
(manufactured by FUJIMI INCORPORATED)

Dilution of the polishing composition: 1:2 deionized water

Supply rate of the

polishing composition: 100 cc/min

Polishing pad: Surfin 018-3

(manufactured by FUJIMI

5 INCORPORATED)

Treating pressure: 80 g/cm<sup>2</sup>

Platen rotational speed: 60 rpm

Polishing time: 5 minutes

Polishing tests

10 Then, using the test samples of Examples 1 to 32 and  
Comparative Examples 1 to 13 and the substrates polished  
by the first step polishing, polishing tests were carried  
out. The polishing conditions were as follows. Further,  
the surface roughness (Ra) of the substrates was measured  
15 by means of an optical surface roughness meter MicroXam  
[X50] (manufactured by Phase Shift, U.S.A.), and the  
value whereby measured was recorded.

Polishing conditions

Polishing machine: Double side polishing  
20 machine

Object to be polished: 3.5" electroless Ni-P  
Substrate (already  
polished in the first  
step, surface roughness:  
25 Ra=12Å)

Number of polished sheets: (two sheets/carrier) × 5  
carriers × 2 batches = 20

sheets

polishing pad: BELLATRIX N0058  
(manufactured by Kanebo,  
Ltd.)

5 Treating pressure: 100 g/cm<sup>2</sup>  
Platen rotational speed: 40 rpm  
Dilution of the polishing  
composition: stock solution  
supply rate of the  
10 polishing composition: 50 cc/min  
polishing time: 10 minutes

After the polishing, the substrate was sequentially  
cleaned and dried, and the weight reduction of the  
substrate by the polishing was measured. The  
15 measurements were carried out with respect to all of the  
polished 20 sheets, and the stock removal rate was  
obtained from the average. The evaluation standards were  
as follows.

◎: at least 0.1 μm/min  
20 ◎: at least 0.05 μm/min and less than 0.01 μm/min  
×: less than 0.05 μm/min

Then, the front and rear sides of all substrates  
after polishing, were inspected by visual observation  
under a spotlight (500,000 lux, manufactured by Yamada  
25 Kogaku Kogyo K.K.) in a dark room, and the number of  
scratches was counted, and from the average, the number  
of scratches per side was obtained. The evaluation

standards were as follows.

◎: less than 10 lines/side

○: at least 10 lines/side and less than 20  
lines/side

5      ×: at least 20 lines/side

The evaluation results with respect to the stock  
removal rate and formation of scratches, were as shown in  
Table 1.

Table 1

	Content of colloidal silica (wt%)	Organic acid		Oxidizing agent		pH	Stock removal rate	Scratches
		Type	Content (wt%)	Type	Content (wt%)			
Ex. 1	1.3	Malic acid	0.5	Hydrogen peroxide	1.6	2.4	⊙	⊙
Ex. 2	4.5	Malic acid	0.5	Hydrogen peroxide	1.6	2.9	⊙	⊙
Ex. 3	8.8	Malic acid	0.5	Hydrogen peroxide	1.6	3.0	⊙	⊙
Ex. 4	4.5	Malic acid	1.0	Hydrogen peroxide	1.6	2.6	⊙	⊙
Ex. 5	4.5	Malic acid	0.5	Hydrogen peroxide	0.6	2.9	⊙	⊙
Ex. 6	4.5	Malic acid	0.5	Hydrogen peroxide	0.2	2.9	⊙	⊙
Ex. 7	4.5	Maleic acid	0.25	Hydrogen peroxide	1.6	2.3	⊙	⊙
Ex. 8	4.5	Maleic acid	0.5	Hydrogen peroxide	1.6	2.1	⊙	⊙
Ex. 9	8.8	Maleic acid	0.25	Hydrogen peroxide	1.6	2.4	⊙	⊙
Ex. 10	4.5	Lactic acid	1.0	Hydrogen peroxide	1.6	2.7	⊙	⊙
Ex. 11	8.8	Lactic acid	1.0	Hydrogen peroxide	1.6	2.8	⊙	⊙
Ex. 12	4.5	Lactic acid	0.5	Hydrogen peroxide	1.6	3.0	⊙	⊙
Ex. 13	4.5	Lactic acid	1.0	Hydrogen peroxide	0.2	2.7	⊙	⊙
Ex. 14	4.5	Acetic acid	1.0	Hydrogen peroxide	1.6	3.3	⊙	⊙
Ex. 15	8.8	Acetic acid	1.0	Hydrogen peroxide	1.6	3.4	⊙	⊙
Ex. 16	4.5	Acetic acid	0.5	Hydrogen peroxide	1.6	3.0	⊙	⊙
Ex. 17	4.5	Acetic acid	1.0	Hydrogen peroxide	0.2	3.3	⊙	⊙
Ex. 18	4.5	Citric acid	1.0	Hydrogen peroxide	0.3	2.5	⊙	⊙
Ex. 19	4.5	Citric acid	1.0	Hydrogen peroxide	3.1	2.6	⊙	⊙
Ex. 20	8.8	Citric acid	1.0	Hydrogen peroxide	1.6	2.5	⊙	⊙
Ex. 21	4.5	Succinic acid	1.0	Hydrogen peroxide	1.6	3.0	⊙	⊙
Ex. 22	4.5	Malonic acid	1.0	Hydrogen peroxide	1.6	2.1	⊙	⊙
Ex. 23	4.5	Glycolic acid	1.0	Hydrogen peroxide	1.6	2.5	⊙	⊙
Ex. 24	4.5	Adipic acid	1.0	Hydrogen peroxide	1.6	3.3	⊙	⊙
Ex. 25	4.5	Ascorbic acid	1.0	Hydrogen peroxide	1.6	2.1	⊙	⊙
Ex. 26	4.5	Itaconic acid	1.0	Hydrogen peroxide	1.6	2.9	⊙	⊙
Ex. 27	4.5	Iminodiacetic acid	1.0	Hydrogen peroxide	1.6	2.5	⊙	⊙
Ex. 28	4.5	Glyoxylic acid	1.0	Hydrogen peroxide	1.6	2.6	⊙	⊙
Ex. 29	4.5	Formic acid	1.0	Hydrogen peroxide	1.6	2.6	⊙	⊙
Ex. 30	4.5	Acrylic acid	1.0	Hydrogen peroxide	1.6	3.1	⊙	⊙
Ex. 31	4.5	Crotonic acid	1.0	Hydrogen peroxide	1.6	3.5	⊙	⊙
Ex. 32	4.5	Nicotinic acid	1.0	Hydrogen peroxide	1.6	3.9	⊙	⊙



Table 2

	Content of colloidal silica (wt%)	Organic acid		Oxidizing agent		pH	Stock removal rate	Scratches
		Type	Content (wt%)	Type	Content (wt%)			
Comp. Ex. 1	4.5	Malic acid	1.0	-	-		x	x
Comp. Ex. 2	4.5	Maleic acid	1.0	-	-		x	x
Comp. Ex. 3	4.5	Lactic acid	1.0	-	-		x	x
Comp. Ex. 4	4.5	Acetic acid	1.0	-	-		x	x
Comp. Ex. 5	4.5	Glutamic acid	1.0	Hydrogen peroxide	1.6	7.9	x	x
Comp. Ex. 6	4.5	Malic acid	1.0	Potassium chlorate	2.0	2.6	○	x
Comp. Ex. 7	4.5	Malic acid	1.0	Potassium persulfate	2.0	2.6	○	x
Comp. Ex. 8	4.5	Sodium malate	1.0	Hydrogen peroxide	1.6	9.0	x	x
Comp. Ex. 9	4.5	Disodium maleate	1.0	Hydrogen peroxide	1.6	8.8	x	x
Comp. Ex. 10	13.9	Iron (III) nitrate	3.0	-	-	2.0	⊙	x
Comp. Ex. 11	13.9	Iron (III) nitrate	3.0	Hydrogen peroxide	1.6	2.0	⊙	x
Comp. Ex. 12	4.5	Ammonium ion (III) citrate	1.0	-	-	7.5	x	x
Comp. Ex. 13	4.5	Ammonium ion (III) citrate	1.0	Hydrogen peroxide	1.6	6.3	⊙	x

From the results of Examples 1 to 32 in Table 1, it is evident that with the polishing composition of the present invention, a high stock removal rate can be obtained. Further, from the results of Examples 1 to 32 and Comparative Examples 1 to 4, it is evident that the polishing composition of the present invention is capable of providing a high polishing performance.

Further, from the results of Examples 1 to 32 and Comparative Examples 5, 12 and 13, the polishing composition of the present invention comprising at least one organic acid selected from the group consisting of malic acid, maleic acid, lactic acid, acetic acid, citric acid, succinic acid, malonic acid, glycolic acid, adipic acid, ascorbic acid, itaconic acid, iminodiacetic acid, glyoxylic acid, formic acid, acrylic acid, crotonic acid and nicotinic acid, is capable of preventing formation of scratches, as compared with the composition comprising glutamic acid as an organic acid other than the above or ammonium iron (III) citrate as an iron salt of an organic acid.

Further, from the results of Example 4 and Comparative Examples 6 and 7, it is evident that the polishing composition of the present invention has a high stock removal rate and is capable of preventing formation of scratches, as compared with the composition containing potassium chlorate or potassium persulfate which is an oxide containing metal ions.

Further, from the results of Examples 1 to 9 and Comparative Examples 8 and 9, it is evident that the polishing composition of the present invention containing an organic acid, has a high stock removal rate and is  
5 capable of preventing formation of scratches, as compared with the composition containing a salt of an organic acid.

And, from the results of Examples 1 to 26 and Comparative Examples 10 to 13, it is evident that the  
10 polishing composition of the present invention is capable of preventing formation of scratches, as compared with the composition containing a conventional iron compound and the composition which further contains an oxidizing agent.

15 Further, the substrate polished by the test sample in each of Examples 1 to 32 and Comparative Examples 1 to 13, had a small surface roughness, which was not a problematic level.

As described in the foregoing, it has been found  
20 that the polishing composition of the present invention has a high stock removal rate, is capable of suppressing formation of scratches and capable of reducing the surface roughness and can provide a high stock removal rate.

25 Thus, the polishing composition of the present invention has a high stock removal rate, is capable of suppressing formation of scratches and capable of

reducing the surface roughness and can bring about a high stock removal rate.

Further, by using the polishing composition of the present invention, a high stock removal rate can be  
5 provided, formation of scratches can be suppressed, and the surface roughness can be made small, and a substrate for a memory hard disk having an excellent polished surface, can be produced efficiently.

Further, by subjecting a substrate for a memory hard  
10 disk which has been subjected to preliminary polishing at least once and has a surface roughness Ra of at most 30 Å before finish polishing, to finish polishing by means of the polishing composition of the present invention, it is possible to obtain a substrate for a memory hard disk  
15 having an excellent finished surface.

CLAIMS:

1. A polishing composition for a memory hard disk, which comprises water, silicon dioxide, an oxidizing agent, and at least one organic acid selected from the group  
5 consisting of malic acid, maleic acid, lactic acid, acetic acid, citric acid, succinic acid, malonic acid, glycolic acid, adipic acid, ascorbic acid, itaconic acid, iminodiacetic acid, glyoxylic acid, formic acid, acrylic acid, crotonic acid, nicotinic acid, citraconic acid and  
10 tartaric acid, and which has a pH of at least 1 and less than 7 and contains substantially no metal ions.
2. The polishing composition for a memory hard disk according to Claim 1, wherein the content of metal ions is at most 500 ppm.
- 15 3. The polishing composition for a memory hard disk according to Claim 1 or 2, wherein the silicon dioxide is colloidal silica.
4. The polishing composition for a memory hard disk according to any one of Claims 1 to 3, wherein the  
20 oxidizing agent is at least one member selected from the group consisting of chloric acid, perchloric acid, sulfuric acid, persulfuric acid, nitric acid, pernitric acid, hydrogen peroxide, iodic acid and periodic acid.
5. The polishing composition for a memory hard disk  
25 according to Claim 4, wherein the oxidizing agent is hydrogen peroxide.
6. The polishing composition for a memory hard disk

according to any one of Claims 1 to 5, wherein the organic acid is at least one member selected from the group consisting of malic acid, maleic acid, lactic acid, itaconic acid and acetic acid.

5 7. The polishing composition for a memory hard disk according to any one of Claims 1 to 6, wherein the content of silicon dioxide is from 1 to 40%, based on the weight of the polishing composition.

8. The polishing composition for a memory hard disk  
10 according to any one of Claims 1 to 7, wherein the content of the oxidizing agent is from 0.1 to 5%, based on the weight of the polishing composition.

9. The polishing composition for a memory hard disk according to any one of Claims 1 to 8, wherein the  
15 content of the organic acid is from 0.01 to 10%, based on the weight of the polishing composition.

10. The polishing composition for a memory hard disk according to any one of Claims 1 to 9, which has a pH of at least 2 and less than 4.

20 11. A method for producing a memory hard disk, which comprises polishing a Ni-P disk having a surface roughness Ra of at most 30Å, by means of the polishing composition as defined in any one of Claims 1 to 9.

12. A method for producing a memory hard disk, which  
25 comprises preliminarily carrying out preliminary polishing at least once to obtain a substrate for a memory hard disk having a surface roughness Ra of at most

15Å and polishing the substrate for final polishing by means of the polishing composition as defined in any one of Claims 1 to 9.

13. A polishing composition for a memory hard disk substantially as any one of the compositions of Examples 1 to 32 herein.

14. A method of polishing a memory hard disk substrate substantially as described in any one of Examples 1 to 32 herein.



INVESTOR IN PEOPLE

Application No: GB 0206036.6  
Claims searched: 1-14

Examiner: Dr Annabel Ovens  
Date of search: 28 August 2002

## Patents Act 1977 Search Report under Section 17

### Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.T): C4V

Int Cl (Ed.7): C09G 1/02; C09K 3/14

Other: Online: PAJ, EPODOC, WPI

### Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
X, E	GB 2354769 A (FUJIMI) see page 8 line 26-page 9 line 13 and Table 1	1-5 and 7-14
X, E	EP 1125999 A1 (FUJIMI) see page 3 lines 44-54 and compositions 3, 5, 7 and 9-15 in Tables 1 and 2	1-14
X	EP 0811665 A2 (CABOT) see page 2 line 57-page 3 line 6, page 3 lines 34-36, page 4 lines 32-36 and 55-58 and Examples	1-14
X, E	US 20010018270 (TSUCHIYA ET AL.) see page 2 paragraphs 19-22, page 3 paragraphs 39-41 and Table 4	1-14

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.